

CLAIMS

We claim:

1. A mesoporous silica film prepared from a surfactant containing solution, having a dielectric constant less than 3 that has both a relative stability and an absolute stability in a humid atmosphere, a film thickness from about 0.1 μm to about 1.5 μm , and an average pore diameter less than or equal to about 20 nm.
2. The mesoporous silica film as recited in claim 1, wherein said average pore diameter is less than or equal to about 10 nm.
3. The mesoporous silica film as recited in claim 1, wherein said thickness has a standard deviation less than +/- 5%.
4. The mesoporous silica film as recited in claim 1, wherein a porosity of said mesoporous silica film is disordered.
5. A mesoporous silica film having a thickness from about 0.1 μm to about 1.5 μm and a standard deviation about said thickness, wherein said standard deviation is less than +/- 5%.
6. The mesoporous silica film as recited in claim 5, wherein a dielectric constant of said mesoporous silica film is less than 3.
7. The mesoporous silica film as recited in claim 5, having a dielectric constant with a relative stability and an absolute stability.
8. The mesoporous silica film as recited in claim 5, having an average pore size less than or equal to about 20 nm.
9. The mesoporous silica film as recited in claim 5, having a porosity that is disordered.

10. A mesoporous silica film prepared from a surfactant containing solution, comprising a porosity that is disordered, said porosity having an average pore diameter of less than or equal to about 20 nm, and a film thickness from about 0.1 μm to about 1.5 μm .

11. The mesoporous silica film as recited in claim 10, having a dielectric constant less than 3, said dielectric constant having both a relative stability and an absolute stability.

- 10 12. A method of making a mesoporous film comprising the steps of:
- (a) combining a silica precursor with an aqueous solvent, a catalyst and a surfactant into a precursor solution;
 - (b) spin coating said precursor solution into a templated film;
 - (c) removing said aqueous solvent, said catalyst and said surfactant from said templated film and forming a hydroxylated film with disordered porosity; and
 - (d) dehydroxylating said hydroxylated film and obtaining said mesoporous film.
- 20 13. The method as recited in claim 12, wherein said surfactant is a polyoxyethylene ether surfactant.
14. The method as recited in claim 13, wherein said polyoxyethylene ether surfactant is $\text{C}_{12}\text{H}_{25}(\text{CH}_2\text{CH}_2\text{O})_{10}\text{OH}$ also known as $\text{C}_{12}\text{EO}_{10}$ or 10 lauryl ether; $\text{C}_{16}\text{H}_{33}(\text{CH}_2\text{CH}_2\text{O})_{10}\text{OH}$ also known as $\text{C}_{16}\text{EO}_{10}$ or 10 cetyl ether; $\text{C}_{18}\text{H}_{37}(\text{CH}_2\text{CH}_2\text{O})_{10}\text{OH}$ also known as $\text{C}_{18}\text{EO}_{10}$ or 10 stearyl ether; $\text{C}_{12}\text{H}_{25}(\text{CH}_2\text{CH}_2\text{O})_4\text{OH}$ also known as C_{12}EO_4 or 4 lauryl ether; $\text{C}_{16}\text{H}_{33}(\text{CH}_2\text{CH}_2\text{O})_2\text{OH}$ also known as C_{16}EO_2 or 2 cetyl ether or combinations thereof.
- 30 15. The method as recited in claim 12, wherein said surfactant is in combination with a chemical agent selected from the group of a second surfactant, smaller hydrophilic molecular compounds, and with organic co-solvents.

16. The method as recited in claim 15, wherein said second surfactant is selected from the group consisting of non-ionic surfactant, cationic surfactant, anionic surfactant, amphoteric surfactant and combinations thereof.

5 17. The method as recited in claim 16, wherein said cationic surfactant is an ammonium-based surfactant.

10 18. The method as recited in claim 15, wherein said smaller hydrophilic molecular compounds are selected from the group consisting of glycerol, propylene glycol, and ethylene glycol.

15 19. The method as recited in claim 15, wherein said organic co-solvents are selected from the group consisting of mesitylene, octane and combinations thereof.

20 20. The method as recited in claim 12, wherein said silica precursor is selected from the group consisting of tetraethyl orthosilicate (TEOS), tetramethyl orthosilicate (TMOS), methyl triethoxysilane, phenyl triethoxy silane, dimethyl dimethoxy silane ethyl triethoxysilane, and combinations thereof.

25 21. The method as recited in claim 12, wherein said aqueous solvent comprises ethanol and water.

22. The method as recited in claim 12, wherein said acid is selected from the group consisting of inorganic acid, organic acid and combinations thereof.

25 23. The method as recited in claim 12, wherein said precursor solution includes at least one other surfactant.

30 24. The method as recited in claim 12, wherein said precursor solution includes at least one smaller hydrophilic molecular compound.

25. The method as recited in claim 12 wherein said precursor solution includes at least one organic co-solvent.

26. The method as recited in claim 23, wherein said at least one other surfactant is selected from the group consisting of non-ionic surfactant, cationic surfactant, anionic surfactant, amphoteric surfactant and combinations thereof.

5 27. The method as recited in claim 24, wherein said at least one smaller hydrophilic molecular compound is selected from the group consisting of glycerol, propylene glycol, ethylene glycol and combinations thereof.

10 28. The method as recited in claim 25, wherein said at least one organic co-solvent is selected from the group consisting of mesitylene, octane and combinations thereof.

15 29. The method as recited in claim 12, wherein dehydroxylating occurs in the presence of a silicon-based organic compound in the vapor phase.

30. The method as recited in claim 29, wherein the silicon-based organic compound is a silane.

20 31. The method as recited in claim 30, wherein the silane is selected from the group consisting of trimethyl iodosilane, trimethyl chlorosilane, dimethyl dimethoxy silane, demethyl dichloro silane, hexaphenyl disilazane, diphenyl tetramethyl silazane and hexamethyl disilazane .

25 32. A method of making a mesoporous film with a surfactant containing solution, the method comprising the steps of:

- (a) combining a silica precursor with an aqueous solvent, a catalyst and a surfactant that is a polyoxethylene ether surfactant into a precursor solution;
- (b) spin coating said precursor solution into a templated film;
- (c) removing said aqueous solvent, said catalyst and said surfactant forming a hydroxylated film having porosity; and
- (d) dehydroxylating said hydroxylated film and obtaining said mesoporous film.

33. The method as recited in claim 32, wherein said polyoxyethylene ether surfactant is $C_{12}H_{25}(CH_2CH_2O)_{10}OH$ also known as $C_{12}EO_{10}$ or 10 lauryl ether; $C_{16}H_{33}(CH_2CH_2O)_{10}OH$ also known as $C_{16}EO_{10}$ or 10 cetyl ether; $C_{18}H_{37}(CH_2CH_2O)_{10}OH$ also known as $C_{18}EO_{10}$ or 10 stearyl ether; $C_{12}H_{25}(CH_2CH_2O)_4OH$ also known as $C_{12}EO_4$ or 4 lauryl ether; $C_{16}H_{33}(CH_2CH_2O)_2OH$ also known as $C_{16}EO_2$ or 2 cetyl ether or combinations thereof.

34. The method as recited in claim 32, wherein said porosity is disordered as indicated by an absence of an x-ray diffraction peak in the range of 2 to 6 degrees 2-theta.

35. The method as recited in claim 32, wherein said porosity is disordered, lacking a regular geometric arrangement of pores, and the pore structure is characterized by an x-ray diffraction peak between about 0.75 and about 2 degrees 2-theta.

36. The method as recited in claim 32, wherein said precursor solution includes at least one other surfactant.

37. The method as recited in claim 32, wherein said precursor solution includes at least one smaller hydrophilic molecular compound.

38. The method as recited in claim 32, wherein said precursor solution includes at least one organic co-solvent.

39. The method as recited in claim 32, wherein said precursor solution includes an agent selected from the group consisting of a second surfactant, a smaller hydrophilic molecular compound, an organic co-solvent and combinations thereof.

40. The method as recited in claim 36, wherein said at least one other surfactant is selected from the group consisting of non-ionic surfactant, cationic surfactant, anionic surfactant, amphoteric surfactant and combinations thereof.

41. The method as recited in claim 37, wherein said at least one smaller hydrophilic molecular compound is selected from the group consisting of glycerol, propylene glycol, ethylene glycol and combinations thereof.

42. The method as recited in claim 38, wherein said at least one organic co-solvent is selected from the group consisting of mesitylene, octane and combinations thereof.

43. The method as recited in claim 39, wherein said second surfactant is selected from the group consisting of non-ionic surfactant, cationic surfactant, anionic surfactant, amphoteric surfactant and combinations thereof.

44. ~~The method as recited in claim 39, wherein said smaller hydrophilic molecular compound is selected from the group consisting of glycerol, propylene glycol, ethylene glycol and combinations thereof.~~

45. The method as recited in claim 39, wherein said organic co-solvent is selected from the group consisting of mesitylene, octane and combinations thereof.

46. The method as recited in claim 32 wherein said silica precursor is selected
from the group consisting of tetraethyl orthosilicate (TEOS), tetramethyl orthosilicate,
methyl triethoxysilane, phenyl triethoxy silane, dimethyl dimethoxy silane and
combinations thereof.

47. The method as recited in claim 32 wherein said aqueous solvent includes ethanol.

48. The method as recited in claim 32, wherein said catalyst is selected from the group consisting of inorganic acid, organic acid and combinations thereof.

49. The method as recited in claim 48, wherein said organic acid is carboxylic acid selected from the group consisting of methanoic acid (formic acid), ethanoic acid (acetic acid), ethandioic acid (oxalic acid), butanoic acid (butyric acid), and combinations thereof.

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50. The method as recited in claim 32, wherein dehydroxylating occurs in the presence of a silicon-based organic compound in the vapor phase.

10 51. The method as recited in claim 50, wherein the silicon-based organic compound is a silane

15 52. The method as recited in Claim 51, wherein the silane is selected from the group consisting of trimethyl iodosilane, trimethyl chlorosilane, dimethyl dichloro silane, hexaphenyl disilazane, diphenyl tetramethyl silazane and hexamethyl disilazane.

20 53. A mesoporous silica film made by the method of claim 32, comprising: a disordered porosity, lacking a regular geometric arrangement of pores, and characterized by an x-ray diffraction peak between about 0.75 and about 2 degrees 2-theta;

a dielectric constant less than 3.0 that is stable;
a film thickness from about 0.1 μm to about 1.5 μm ; and
an average pore diameter less than or equal to about 20 nm.

25 54. A mesoporous silica film made by the method of claim 32, comprising: a disordered porosity as indicated by an absence of an XRD peak in the range from 2 to 6 degrees 2-theta;

a dielectric constant less than 3.0 that is stable;
a film thickness from about 0.1 μm to about 1.5 μm ; and
an average pore diameter less than or equal to about 20 nm.

30 55. A mesoporous film made by the method of claim 12, comprising:
a dielectric constant less than 3.0 that is stable;

a film thickness from about 0.1 μm to about 1.5 μm ; and
an average pore diameter less than or equal to about 20 nm.

56. A method of making a mesoporous film comprising the steps of:
- 5 (a) combining a silica precursor with an aqueous solvent, an acid and a polyoxyethylene ether surfactant into a precursor solution;
 - (b) spin-coating said precursor solution into a templated film;
 - (c) removing said aqueous solvent, said acid and said surfactant forming a hydroxylated film; and
 - 10 (d) dehydroxylating said hydroxylated film and obtaining said mesoporous film.
57. The method as recited in claim 56, wherein said polyoxyethylene ether surfactant is $\text{C}_{12}\text{H}_{25}(\text{CH}_2\text{CH}_2\text{O})_{10}\text{OH}$ also known as $\text{C}_{12}\text{EO}_{10}$ or 10 lauryl ether;
- 15 $\text{C}_{16}\text{H}_{33}(\text{CH}_2\text{CH}_2\text{O})_{10}\text{OH}$ also known as $\text{C}_{16}\text{EO}_{10}$ or 10 cetyl ether; $\text{C}_{18}\text{H}_{37}(\text{CH}_2\text{CH}_2\text{O})_{10}\text{OH}$ also known as $\text{C}_{18}\text{EO}_{10}$ or 10 stearyl ether; $\text{C}_{12}\text{H}_{25}(\text{CH}_2\text{CH}_2\text{O})_4\text{OH}$ also known as C_{12}EO_4 or 4 lauryl ether; $\text{C}_{16}\text{H}_{33}(\text{CH}_2\text{CH}_2\text{O})_2\text{OH}$ also known as C_{16}EO_2 or 2 cetyl ether or combinations thereof.
- 20 58. The method as recited in claim 57, wherein said polyoxyethylene ether surfactant is in combination with other small surfactants, with smaller hydrophilic molecules, and with organic co-solvents.
- 25 59. The method as recited in claim 58, wherein said small surfactants are ammonium-based surfactants.
60. The method as recited in claim 59, wherein said ammonium-based surfactants are cetyl trimethyl ammonium chloride.
- 30 61. The method as recited in claim 58, wherein said smaller hydrophilic molecules are selected from the group consisting of glycerol, propylene glycol, and ethylene glycol.

62. The method as recited in claim 58, wherein said organic co-solvents are selected from the group consisting of mesitylene and octane.
63. The method as recited in claim 56, wherein said silica precursor is 5 tetraethyl orthosilicate (TEOS).
64. The method as recited in claim 56, wherein said aqueous solvent comprises ethanol and water.
- 10 65. The method as recited in claim 56, wherein said acid is hydrochloric acid.
- 15 66. A mesoporous film having a dielectric constant less than 2.5, a film thickness from about 0.2 μm to about 1.5 μm , and an average pore diameter less than or equal to about 5 nm.
67. A mesoporous film having a thickness from about 0.2 μm to about 1.5 μm and a standard deviation about said thickness that is less than +/- 5%.
- 20 68. A mesoporous silica film prepared from a surfactant containing solution, having a dielectric constant less than 3 that has both a relative stability and an absolute stability in a humid atmosphere, a film thickness from about 0.1 μm to about 1.5 μm , an average pore diameter less than or equal to about 20 nm, and a porosity that is disordered.
- 25 69. The mesoporous silica film as recited in claim 68, wherein disordered is indicated by the absence of an X-ray diffraction peak in the range of about 2 to about 6 degrees 2-theta.
70. The mesoporous silica film as recited in claim 68, wherein 30 disordered porosity is characterized by an X-ray diffraction peak between about 0.75 and about 2 degrees 2-theta.

71. A method of making a mesoporous film comprising the steps of:

- (a) combining a silica precursor with an aqueous solvent, a catalyst and a surfactant into a precursor solution;
- (b) spin coating said precursor solution into a templated film;
- 5 (c) removing said aqueous solvent, said catalyst and said surfactant from said templated film and forming a hydroxylated film; and
- (d) dehydroxylating said hydroxylated film with a gaseous silicon-based organic compound and obtaining said mesoporous film.

10 72. The method of claim 71, wherein the silicon-based organic compound is a silane.

15 73. The method of claim 71, wherein said dehydroxylation of said film occurs in alternating exposures of said film to a vacuum and to the gaseous silane.

74. The method of claim 71, wherein said silane is selected from the group consisting of trimethyl iodosilane, trimethyl chlorosilane, dimethyl dimethoxy silane, dimethyl dichloro silane, hexaphenyl disilazane, diphenyl tetramethyl silazane and hexamethyl disilazane.

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